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(54) Title: DETERGENT COMPOSITIONS

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(57) Abstract

A detergent composition suitable for washing fabrics is in the form of a stable oil-in-water microemulsion and comprises an organic surfactant system, wholly or predominantly consisting of short-chain ethoxylated nonionic surfactant having an average alkyl chain length of less than C12 and containing a substantial amount of C10 material, a non-aqueous solvent such as hexadecane, and optionally a water-soluble detergency builder. The composition can be used both for pre-wash treatment and as a main wash detergent.

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DETERGENT COMPOSITIONS

TECHNICAL FIELD

The present invention relates to detergent compositions containing a surfactant and a solvent in the form of an oil-in-water microemulsion.

BACKGROUND AND PRIOR ART

Liquid detergent and cleaning compositions in the form of microemulsions, both oil-in-water and water-in-oil, have been disclosed in the prior art.

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EP 137 616A (Procter & Gamble) discloses liquid detergent compositions prepared from conventional detersive surfactants and other conventional detergent ingredients, plus a grease-cutting solvent. The compositions contain fatty acids or soaps (5-50 wt%) as detergency builders and are formulated as stable oil-in-water microemulsions. The preferred surfactant systems comprise sulphonate or sulphate type anionic surfactants with minor amounts of ethoxylated nonionic surfactants such as C_{14-15} alcohol ethoxylates (7EO). Detergency builders may be present in amounts of 0.5-15 wt%, citrates being preferred.

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EP 164 467A (Procter & Gamble) discloses laundry detergents and hard surface cleaners comprising oil-in-water microemulsions, containing alkylbenzene and olefin solvents, plus surfactants and substantial amounts of fatty acid soap. The compositions may contain ethoxylated nonionic surfactants, for example, C₁₄₋₁₅ alcohol ethoxylate (7EO). Compositions containing sodium citrate as builder are disclosed.

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In "Evaluation of Textile Detergent Efficiency of Microemulsions in Systems of Water, Nonionic Surfactant and Hydrocarbon at Low Temperature", J Dispersion Science and Technology, 6(5), 523-537 (1985), Marcel Dekker Inc, C Solans, J Garcia Lominguez and S E Friberg describe the use of such microemulsions for washing under conditions of minimum mechanical energy and at low temperatures. The systems studied contain C_{12} alkyl ethoxylate (4EO) nonionic surfactant, water and hexadecane, and optionally small amounts of cosurfactant (sodium dodecyl sulphate), or electrolyte (sodium tripolyphosphate or sodium citrate).

GB 2 194 547A (Colgate-Palmolive) discloses a clear single-phase liquid pre-spotting composition in the form of a microemulsion (oil-in-water or water-in-oil), solution or gel, comprising 10-70 wt% alkane (solvent), 4-60 wt% nonionic surfactant, optional cosurfactants and/or cosolvents, and 1-80 wt% water. It is suggested that builders such as sodium sesquicarbonate might be included, preferably at levels of 5 wt% and above. Unbuilt water-in-oil microemulsions are specifically disclosed which contain mixtures of the short-chain nonionic surfactant Neodol 91-6 in conjunction with a longer-chain (C_{14-15}) ethoxylated nonionic surfactant.

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DEFINITION OF THE INVENTION

The present invention provides a fabric washing detergent composition comprising:

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(i) from 2 to 40 wt% of an organic surfactant system comprising:

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(a) 50-100 wt% of ethoxylated alcohol nonionic surfactant having an average alkyl chain length of less than C_{12} and a content of C_{10} material (based on the alcohol) of at least 45 wt%;

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- (b) optionally up to 50 wt% of co-surfactant other than ethoxylated alcohol nonionic surfactant,
- (ii) from 0.5 to 55 wt% of non-aqueous solvent,

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- (iii) optionally from 0.1 to 5 wt% of water-soluble detergency builder,
- (iv) water and optional minor ingredients to 100 wt%,

wherein the surfactant system (i) and the non-aqueous 25 solvent (ii) together with water form a stable oil-in-water microemulsion.

DETAILED DESCRIPTION OF THE INVENTION

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The present inventors have now discovered that detergent compositions in oil-in-water microemulsion form, formulated with specific nonionic surfactants having short alkyl chains, are capable of sufficiently rapid cleaning and stain removal to render them useful as pretreatment products as well as main wash products.

Rapidity of cleaning effect is of critical importance for a pretreatment product which is required to work within a short time period. The present invention enables detergent compositions to be formulated which are highly effective main wash products and yet which also offer a potent pretreatment facility.

The compositions are also suitable for use in machine washing employing automatic dosing systems, for example, as described and claimed in US 4 489 455 (Procter & Gamble). This patent describes and claims apparatus and process for washing textiles based on utilising strictly limited or controlled quantities of an aqueous wash liquor, ranging from (at least) just enough to be distributed evenly and completely over the whole wash load, to (at most) about five times the dry weight of the washload.

In the compositions of the invention, which are preferably liquid, the ethoxylated nonionic surfactant and the solvent are so chosen, and are present in amounts such that, together with water, they form a stable oil-in-water microemulsion in which the solvent is within the micelles of the surfactant.

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The ethoxylated nonionic surfactant

The ethoxylated nonionic surfactant has an average alkyl chain length which is less than 12 carbon atoms. Preferably the average alkyl chain length is within the range of from 9 to 11 carbon atoms, and most preferably the average alkyl chain length is about C_{10} .

The ethoxylated nonionic surfactant is also characterised by a high content of C_{10} material: at least 45 wt%, preferably at least 50 wt% and most preferably at least 70 wt% (all based on the alcohol).

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The remainder of the ethoxylated nonionic surfactant may be of predominantly shorter or longer chain length, but advantageously the total content of C_{10} and shorter-chain material is at least 60 wt%, and more preferably at least 75 wt% (all based on the alcohol).

Suitable materials are the Novel (Trade Mark) 1012 series ex Vista, which are narrow-range-ethoxylated materials consisting mainly of C_{10} chains, available in various average degrees of ethoxylation. The chain length distribution of these materials (based on the alcohol) is typically C_{10} 84 \pm 4%, C_{12} 8.5 \pm 2%, C_{14} 6.5 \pm 2%.

A class of broader-range-ethoxylated materials suitable for use in the invention is the Dobanol (Trade Mark) 91 series ex Shell, which consist mainly of C_9 , C_{10} and C_{11} chains. The chain length distribution of these materials (based on the alcohol) is typically C_9 18%, C_{10} 50%, C_{11} 32%.

Other short chain nonionic surfactants are described in detail in WO 94 11487A (Unilever). These include the Lialet (Trade Mark) 91 series ex Enichem, the Symperonic (Trade Mark) 91 series ex ICI, and a C₁₀ Inbentin (Trade Mark) material ex Kolb.

Commercial ethoxylated nonionic surfactants are generally mixtures containing a spread of chain lengths about an average value. If desired, a mixture of two or more commercial materials may be used provided that the overall average chain length of all nonionic surfactant present is less than C_{12} and provided that sufficient C_{10} material is present in the overall mixture.

The average degree of ethoxylation may suitably range from 2 to 8, and preferably from 2 to 6, in order to give optimum HLB (hydrophilic-lipophilic balance) values corresponding to optimum oily soil detergency.

The HLB value suitably ranges from 8 to 14, preferably from 8 to 12.5, and more preferably from 9 to 10.

the optional cosurfactant

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If desired, a cosurfactant which is not an ethoxylated alcohol may be present, provided that at least 50 wt% of the surfactant system is constituted by ethoxylated nonionic surfactant. The co-surfactant may be, for example, a nonionic surfactant other than an ethoxylated alcohol, or an anionic sulphate or sulphonate type detergent, such as alkylbenzene sulphonate or primary alcohol sulphate. It is generally preferred that the surfactant system should contain not more than 40 wt% of anionic surfactant.

The surfactant system as a whole constitutes from 2 to 40 wt% of the composition, preferably from 5 to 40 wt%, more preferably from 5 to 30 wt% and advantageously from 5 to 25 wt%, of the composition.

The non-aqueous solvent

The non-aqueous solvent, which constitutes from 0.5 to 55 wt%, preferably from 0.5 to 20 wt%, of the composition, may be any solvent valuable in the removal of oily soil which exhibits a sufficiently low interfacial tension towards the ethoxylated nonionic surfactant to form a stable oil-in-water microemulsion.

The solvent may range from wholly non-polar paraffinic materials, for example, alkanes, to more polar materials such as esters. Preferred solvents are C_{12-16} alkanes, for example, dodecane, tetradecane and hexadecane, hexadecane being especially preferred.

When the solvent is an alkane, the optimum amount present depends on the chain length. For hexadecane, from 1 to 20 wt%, preferably from 5 to 15 wt% and more preferably from 7.5 to 15 wt%, is suitable; for tetradecane, 15 to 30 wt% is preferred, and for dodecane, 25 to 55 wt% is preferred.

The weight ratio of non-aqueous solvent (alkane) to ethoxylated nonionic surfactant is also dependent on chain length. For hexadecane, it lies suitably within the range of from 0.5:1 to 2:1, and is advantageously about 1:1.

The detergency builder

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It has been found that the detergency of the microemulsion system, as compared to the detergency of the same amount of surfactant alone, may be significantly increased if there is also present a detergency builder. The amount of builder that can be incorporated without destabilising the microemulsion is not, however, unlimited. Suitably, a builder may be present in an amount of from 0.1 to 5 wt%, preferably from 0.2 to 3 wt%. However, unbuilt compositions are also within the scope of the invention.

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Suitable detergency builders include inorganic builders, for example, sodium tripolyphosphate, and organic builders, for example, sodium citrate.

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However, the elimination of phosphates from detergent compositions has been seen increasingly as environmentally desirable, and citrates, although environmentally impeccable, are not very efficient builders.

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Accordingly, preferred builders for use in the present invention are polymeric polycarboxylate builders, for example, acrylic, maleic and itaconic acid polymers.

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Polymers that may be used include polyacrylates, acrylic/maleic copolymers such as Sokalan (Trade Mark) CP5 and CP7 ex BASF, and the polyvinyl acetate/polyitaconic acid polymers described and claimed in WO 93 23444A (Unilever). These polymers are highly weight-effective builders which can be used in amounts that give significant building without destabilising the microemulsion.

Also suitable are nitrogen-containing monomeric polycarboxylates, for example, nitrilotriacetates and ethylenediamine tetraacetates.

The invention is further illustrated by the following non-limiting Examples, in which parts and percentages are by weight unless otherwise stated.

EXAMPLES

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<u>Detergency</u> assessment

Oily soil detergencies were assessed by measuring the percentage removal of radio-labelled model soils by means of a scintillation counter.

Soiled cloths (5 cm x 5 cm squares of knitted polyester) carrying a mixture of radiolabelled triolein and radiolabelled palmitic acid were prepared as follows. Each cloth was soaked in 0.18 ml of a toluene solution containing 3.33 g 95% triolein (radiolabelled) and 1.67 g 99% palmitic acid (radiolabelled) per 100 ml. The cloths were than allowed to equilibrate for 3 hours.

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Each composition under test was applied to a fabric square at ambient temperature at a level designed to give a liquor to cloth ratio of 1:1. The contact time was varied from 5 to 30 minutes to examine kinetic effects. The cloth was then transferred, using tweezers, to an open bottle containing 15 ml of water (20° French hard) held within a shaker bath maintained at 25°C. The cloth was then rinsed for 2 minutes at a 100 rpm setting of the shaker bath (this gave a gentle to and fro motion to the rinse liquor within the bottle).

After rinsing the liquor was sampled with an automatic pipette (3 x 1 ml aliquots). These aliquots were transferred to plastic vials and were then mixed with 10 ml quantities of scintillator solution prior to being counted on a liquid scintillation counter. The counts (disintegrations per minute, "DPMs") were used to calculate the percentage removal for each soil component under each condition examined. Standards were taken during the initial soiling procedure to give an average figure for the DPMs added in 0.18 ml of soiling solution.

Compositions

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Liquid detergent compositions were prepared to the formulations (in parts by weight) given in the tables that follow. Soil removal (detergency) results are shown after the tables of compositions.

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The compositions of Examples 1 to 6 and Comparative Examples X, P, M and N containing a solvent (hexadecane) were in microemulsion form, while the compositions of Comparative Examples A to E, which did not contain a solvent, were not.

The ingredients used may be identified as follows:

¹Novel (Trade Mark) 1012-52 ex Vista Chemicals: chain length distribution as described previously, 4EO

²Dobanol (Trade Mark) 91-2.5 ex Shell: chain length distribution as described previously, 2.5EO.

These two nonionic surfactants were used together in a weight ratio of 3:1. The combined nonionic surfactant contained about 75 wt% (based on the alcohol) of C₁₀ material, and about 80 wt% (based on the alcohol) of C₁₀ and shorter-chain material. The HLB value was about 9.5.

³Novel (Trade Mark) 1412-4.4EO ex Vista Chemicals: C_{12-14} , 4.4EO.

4Sodium tripolyphosphate.

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20 SEthylenediamine tetracetic acid, tetrasodium salt.

'Copolymer of maleic and acrylic acids, sodium salt: Sokalan (Trade Mark) CP5 ex BASF.

7Copolymer of polyvinyl acetate and itaconic acid, sodium salt, as described and claimed in WO 93 23444A (Unilever).

Example 1. Comparative Examples A. X and Y: no builder

5	Example	1	A	х	Y
-	Nonionic:				
	$C_{10}EO_4^{1}$	7.5	7.5	-	-
10	$C_{9-11}EO_{2.5}^{2}$	2.5	2.5	-	-
	C ₁₂₋₁₄ EO _{4.4} ³	-	-	10.0	10.0
	Hexadecane	10.0	-	10.0	-
15	Water (20°FH)	80.0	90.0	80.0	90.0
		100.0	100.0	100.0	100.0

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The soil removal results for Examples 1 and A containing short-chain nonionic surfactant were as follows:

25	Soak/contact time (minutes)	<u>Soil rem</u> <u>Triolein</u>		moval (%) Palmiti	oval (%) Palmitic acid	
		1	A	1	А	
30	5	32.0	9.8	28.7	21.2	
	10	34.6	11.9	32.6	25.4	
	15	33.7	15.0	30.3	31.6	
	20	33.8	15.1	31.4	30.4	
	30	26.9	14.4	25.6	39.6	
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These results show that, in the absence of builder, in the removal of triolein the microemulsion gave substantially better soil removal throughout the 30-minute test period. The microemulsion also offered a significant kinetic advantage over the non-microemulsion system. With palmitic acid, the advantage was kinetic only.

The corresponding results for Comparative Examples X and Y using longer-chain nonionic surfactant were as follows:

	Soak/contact time		<u>Soil re</u>	moval (%)	
	(minutes)	<u>Triolei</u>	<u>n</u>	<u>Palmiti</u>	c acid
15		x	Y	х	Y
	5	9.4	9.4	29.2	14.2
	10	14.6	9.5	33.1	15.2
20	15	19.7	11.3	34.4	20.5
	20	25.5	13.6	37.3	23.5
	30	31.9	17.0	37.8	29.4

On triolein, the microemulsion system X finally gave results comparable with those obtained from microemulsion system 1, but required the full 30 minutes to do so; the use of short-chain nonionic surfactant clearly gives a significant kinetic advantage. The non-microemulsion system Y was poor, comparable to the non-microemulsion system A.

On palmitic acid, however, the longer-chain nonionic surfactant apparently benefited more than the shorter-chain material from microemulsification.

Example 2. Comparative Examples B. P and O:
sodium tripolyphosphate builder

5	Example	2	В	Р	Q
	Nonionic:				
E.	$C_{10}EO_4^{-1}$	7.5	7.5	-	-
~	$C_{9-11}EO_{2.5}^{2}$	2.5	2.5	-	-
10	$C_{12-14}EO_{4.4}^{3}$	-	-	10.0	10.0
	Hexadecane	10.0	-	10.0	-
	STP ⁴	0.8	0.9	0.8	0.9
15	Water (20°FH)	80.0	90.0	80.0	90.0
		100.8	100.9	100.8	100.9

The soil removal results for Examples 2 and B containing short-chain nonionic surfactant were as follows:

25	Soak/contact time (minutes)	<u>Soil rer</u> <u>Triolein</u>		moval (%) Palmitic acid	
		2	В	2	В
	5	36.2	22.2	49.6	47.3
30	10	50.7	26.3	60.1	50.7
	15	58.7	26.9	60.7	50.0
	20	60.8	28.5	63.6	54.7
	30	63.8	26.1	63.5	55.6

Comparison of these results with those of Example 1 and Comparative Example A shows that both systems performed better in the presence of the highly efficient builder, sodium tripolyphosphate. However, the difference in performance between the microemulsion and the non-microemulsion was substantially increased, very high figures being obtained with the microemulsion. Also, palmitic acid removal was always better with the microemulsion system than with the comparative system.

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The corresponding results for Comparative Examples P and Q using longer-chain nonionic surfactant were as follows:

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Soak/contact time (minutes)	<u>Soil re</u> r <u>Triolein</u>		moval (%) Palmitic acid	
	P	Q	P	Q
20				
5	7.5	20.8	46.5	37.1
10	12.3	26.0	51.6	42.0
15	17.7	31.1	51.7	44.8
20	22.9	33.1	54.8	49.0
25 30	39.5	34.8	55.9	53.8

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On triolein, the microemulsion P gave significantly worse results than the microemulsion 2, and was also slow to reach the maximum value. Of the four systems only 2 gave really high values. The non-microemulsion systems Q and B gave similar results, showing no benefit for the use of short-chain nonionic surfactant in the non-microemulsion system.

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On palmitic acid, little difference was observed between the various systems.

Example 3, Comparative Example C: EDTA builder

	Example	3	С
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	Nonionic:		
	C ₁₀ EO ₄ ¹	7.5	7.5
	$C_{9-11}EO_{2.5}^{2}$	2.5	2.5
	Hexadecane	10.0	_
10	EDTA ⁵	0.8	0.9
+ -	Water (20°FH)	80.0	90.0
		100.8	100.9
15			

Soil removal results were as follows:

20	Soak/contact time	Soil removal (%)			
	(minutes)	<u>Triolein</u>		<u>Palmitic</u>	acid
		3	С	3	
	С				
25					
	5	32.0	16.4	44.5	39.7
	10	45.0	17.0	48.7	40.7
	15	45.6	19.3	46.2	45.7
	20	48.4	21.2	47.4	46.2
30	30	36.0	18.8	44.3	53.4

These results show a similar pattern to that seen with sodium tripolyphosphate builder, but the benefit was smaller. With palmitic acid, only a kinetic advantage was seen.

The following Examples show that much better detergency could be achieved using polymeric builders.

5 Example 4. Comparative Examples D and M: acrylate/maleate copolymer builder

-	Example	4	D	М
10	Nonionic:		7 F	
	C ₁₀ EO ₄ ¹	7.5	7.5	-
	$C_{9-11}EO_{2.5}^{2}$	2.5	2.5	-
	$C_{12-14}EO_{4.4}^{3}$	_	-	10.0
15	Hexadecane	10.0	-	10.0
	AA/MA ⁶	. 0.8	0.9	0.8
	Water (20°FH)	80.0	90.0	80.0
20			⇔	
		100.8	100.9	100.8

The soil removal results were as follows:

25		Triolein			Palmitic acid		
		4	D	М	4	D	М
	5	41.4	12.5	6.0	49.4	27.2	39.0
30	10	53.4	16.5	8.5	54.1	34.3	41.6
	15	56.2	17.1	12.6	56.4	36.4	45.6
	20	59.8	18.6	18.6	59.8	37.4	49.6
	30	58.7	19.2	33.6	62.1	42.7	55.0

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These Examples show the benefits of a microemulsion system and of the use of short-chain nonionic surfactant.

Example 5. Comparative Examples E and N: poly(vinvl acetate/itaconate) builder

5	Example	5	E	N
	Nonionic:			
_	C ₁₀ EO ₄ ¹	7.5	7.5	-
-	C ₉₋₁₁ EO _{2.5} ²	2.5	2.5	-
10	$C_{12-14}EO_{4.4}^{3}$	-	-	10.0
	Hexadecane	10.0	-	10.0
	PVA/IA ⁷	0.8	0.9	0.8
15	Water (20°FH)	80.0	90.0	80.0
		100.8	100.9	100.8

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The soil removal results were as follows:

25			T	<u>Triolein</u> <u>I</u>		Palmitic acid	
		5	E	N	5	E	N
	5	32.3	16.0	3.4	52.3	33.9	41.4
30	10	45.5	17.9	5.3	61.6	41.4	43.8
	15	50.3	20.7	7.9	63.4	45.3	47.1
	20	58.2	20.2	13.6	67.0	47.4	49.5
	30	64.3	20.2	30.1	64.7	48.4	53.8

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These Examples show the benefits of a microemulsion system and of the use of short-chain nonionic surfactant.

Example 6: sodium citrate builder

	Example	6
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	Nonionic:	
	$C_{10}EO_4^1$	7.5
	C ₉₋₁₁ EO ₂₋₅ ²	2.5
-	Hexadecane	10.0
10	Sodium citrate	0.8
-	Water (20°FH)	80.0
		100.8

Soil removal results were as follows:

	Soak/contact time	Soil removal (%)		
	(minutes)	Triolein	Palmitic acid	
20	5	42.0	31.6	
	10	41.9	33.0	
	15	39.7	35.1	
	20	40.8	35.9	
	30	38.3	38.9	

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These results, when compared with Examples 1-5, show some benefit over an unbuilt system, but demonstrate citrate to be a very much less effective builder in these systems than are sodium tripolyphosphate or polymeric builders.

CLAIMS

	1 A fabric washing detergent composition comprising:
5	(i) from 2 to 40 wt% of an organic surfactant system comprising:
10	(a) 50-100 wt% of ethoxylated alcohol nonionic surfactant having an average alkyl chain length of less than C_{12} and a content of C_{10} material (based on the alcohol) of at least 45 wt%;
15	(b) optionally up to 50 wt% of co-surfactant other than ethoxylated alcohol nonionic surfactant,
	(ii) from 0.5 to 55 wt% of non-aqueous solvent,
20	<pre>(iii) optionally from 0.1 to 5 wt% of water-soluble detergency builder,</pre>
	(iv) water and optional minor ingredients to 100 wt%,
25	wherein the surfactant system (i) and the non-aqueous solvent (ii) together with water form a stable oil-in-wate microemulsion.

30 2 A detergent composition as claimed in claim 1, wherein the nonionic surfactant (i)(a) contains at least 70 wt% (based on the alcohol) of C_{10} material.

A detergent composition as claimed in claim 1 or claim 2, wherein the nonionic surfactant (i)(a) contains at least 60 wt% (based on the alcohol) of material having a chain length of C_{10} or less.

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A detergent composition as claimed in any preceding claim, wherein the nonionic surfactant (i)(a) contains at least 80 wt% (based on the alcohol) of material having a chain length of C_{10} or less.

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5 A detergent composition as claimed in any preceding claim, wherein the nonionic surfactant (i)(a) has an HLB value within the range of from 8 to 12.5.

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A detergent composition as claimed in claim 5, wherein the nonionic surfactant (i)(a) has an HLB value within the range of from 9 to 10.

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7 A detergent composition as claimed in any preceding claim, which comprises from 5 to 40 wt% of the surfactant system (i).

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8 A detergent composition as claimed in any preceding claim, wherein the non-aqueous solvent (ii) comprises a C_{12-16} alkane.

A detergent composition as claimed in claim 8, wherein the solvent (ii) comprises hexadecane.

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10 A detergent composition as claimed in claim 9, wherein the hexadecane (ii) is present in an amount of from 0.5 to 20 wt%.

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11 A detergent composition as claimed in claim 10, wherein the hexadecane (ii) is present in an amount of from 5 to 15 wt%.

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- 12 A detergent composition as claimed in any one of claims 9 to 11, wherein the weight ratio of hexadecane (ii) to nonionic surfactant (i)(a) is within the range of from 0.5:1 to 2:1.
- 25 13 A detergent composition as claimed in any preceding claim, which comprises from 0.5 to 3 wt% of detergency builder (iii).

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14 A detergent composition as claimed in any preceding claim, which comprises as detergency builder (iii) sodium tripolyphosphate.

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15 A detergent composition as claimed in any preceding claim, which comprises as detergency builder (iii) a polymeric detergency builder.

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16 A detergent composition as claimed in any preceding claim, wherein the organic surfactant system (i) contains less than 40 wt% of anionic surfactant.

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PCT/EP 95/00990 A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C11D17/00 C11D3/18 C11D1/72 According to International Patent Classification (IPC) or to both national classification and IPC B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) C11D IPC 6 Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Electronic data base consulted during the international search (name of data base and, where practical, search terms used) C. DOCUMENTS CONSIDERED TO BE RELEVANT Relevant to claim No. Citation of document, with indication, where appropriate, of the relevant passages 1,5-12 GB,A,2 194 547 (COLGATE-PALMOLIVE CO.) 9 X March 1988 cited in the application see the whole document CA,A,2 013 431 (PENNZOIL PRODUCTS CO.) 30 1,5-8 X September 1990 see page 8, line 1 - page 9, line 6; claims 1,7-9,19; examples EP,A,0 418 986 (COLGATE-PALMOLIVE CO.) 27 1,5-8,16X March 1991 see page 3, line 41 - page 4, line 24; claims; example 2 1,5-7,16 GB, A, 2 259 518 (SHELL INTERNATIONALE X RESEARCH MAATSCHAPPIJ B.V.) 17 March 1993 see the whole document -/--X Further documents are listed in the continuation of box C. Patent family members are listed in annex. X "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the Special categories of cited documents: "A" document defining the general state of the art which is not considered to be of particular relevance invention "E" earlier document but published on or after the international

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"Y" document of particular relevance; the claimed invention camot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

'&' document member of the same patent family

Date of mailing of the international search report

Date of the actual completion of the international search

- 7. 08. **9**5

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